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Consensus Plan for Soil and Sediment Studies

Coeur d'Alene River Soils and Sediments Bioavailability Studies

U.S. EPA Region 10 (February 2001)

1.0 Introduction

Since the early 1900s, lead poisoning of waterfowl, including mallards (*Anas platyrhynchos*), tundra swans (*Cygnus columbianus*), and Canada geese (*Branta canadensis*) has been reported along the Coeur d'Alene River in Northern Idaho (Chupp and Dalke, 1964; Benson et al., 1976; Blus et al., 1991). Waterfowl can be poisoned by ingestion of spent lead shotgun pellets, fishing weights or lead-contaminated sediments. Previous field and laboratory studies suggest that ingestion of lead-contaminated sediments could be causing waterfowl lead poisoning in the Coeur d'Alene Basin (Heinz et al., 1999; Hoffman et al., 2000; Hoffman et al., 2000).

Addition of an amendment or reagent to soil/sediment is a technique that may reduce the toxicity of the lead to waterfowl. This document describes collaborative field and laboratory studies involving the Idaho Department of Environmental Quality (IDEQ), US Fish and Wildlife Service (USFWS) and the Environmental Protection Agency (EPA) to evaluate the effectiveness of soil amendments in reducing lead bioavailability to waterfowl. The key objectives of these studies is to identify amendment(s) that will reduce lead bioavailability to waterfowl and reduce metal leachability. The IDEQ component of the studies focuses on metal leachability in Coeur d'Alene soils and sediments treated with different candidate amendments. The USFWS portion of the studies focuses on the bioavailability of lead in sediments treated with amendment. The experiments and the methodology are described in this document. There is a companion document which describes the Data Quality Objectives Process for the study (EPA 2001).

This plan for soil and sediment studies is the product of consensus reached by a number of Coeur d'Alene Basin stakeholders. During numerous meetings, teleconferences, and conversations over the last several months there have been discussions of possible reagents and sites to use in evaluating the ability of soil amendments to reduce bioavailability of metals to water fowl. Participants in the discussions included representatives from EPA and their contractors, USFWS, IDEQ, Washington Department of Ecology, CDA Tribe, Spokane Tribe, Ridolfi Engineers, USGS, and others. In addition, representatives of In-Place Inactivation and Natural Ecological

Restoration Technologies (IINERT) Soil-Metals Action Team provided some technical input and review of proposals. Numerous other parties received e-mails providing them with information on the deliberations regarding soil amendments and inviting their participation in the discussions.

1.1 Initial List of Reagents Considered

Following is the list of reagents we discussed in our telephone conference call of October 17.

Granular apatite
Phosphoric acid
Fishbone apatite
Silica encapsulation
Compost
Biosolids/sludge
Iron filings
Iron oxyhydroxides
Zeolites

After the initial review, the extended list of reagents was reduced to the following short list (also discussed in the conference call of October 17, 2000).

Apatite w/wo limestone
Iron oxyhydroxides/limestone
Lime/Limestone
Silica/Zeolites
Organic matter w/wo limestone

Upon further consideration the list has been reduced to:

1. Granular apatite [e.g., fishbone or super triple phosphate (STP)]
2. Liquid apatite (phosphoric acid)

The rationale for reducing the list to granular and phosphoric acid is briefly summarized in the following section. In the FWS bioavailability study, only phosphoric acid will be used since this is the most promising amendment, while in the IDEQ field study granular apatite (fishbone) and phosphoric acid will both be used.

2.0 Rationale for Eliminating/Selecting Reagents

2.1 Rationale for Eliminating Reagents

It is generally preferable to use reagents that operate through mineral formation rather than those that function through adsorption. Adsorption reactions are typically reversible. In the acid environment of the stomach there will be high concentrations of hydronium cations (H_3O^+). The high concentrations ($>10^{-2}$ molar) of hydronium cations (positively charged) will displace positively charged and adsorbed lead cations through mass balance reactions. Even though a given divalent lead cation may adsorb more strongly, the high concentrations of hydronium ions will cause displacement of the adsorbed lead. This results in smaller reductions in bioavailability.

Zeolites and iron oxyhydroxides function by the less preferred mechanism of adsorption. Therefore, these reagents were eliminated from further consideration. Iron filings were eliminated, partly, for the same reason. Iron filings, in the process of being oxidized to ferrous and ferric ions, donate electrons that can potentially reduce other constituents. In the process of oxidizing, the filings develop an iron oxide surface coating that can act as an adsorption surface to remove metals from solution. As mentioned, adsorption was not the preferred metal retention mechanism. The electron donating capacity of iron filings is indirectly considered in the following discussion on organic materials.

Compost and biosolids/sludge were eliminated because it is doubtful how much we can artificially enhance the naturally occurring organic formation. Compost and biosolids/sludge would be most applicable to marshy areas that operate under an anaerobic environment. Plant material is often already decaying/degrading in these areas. The decomposition of dying plant material should help maintain the reducing environment that is conducive to the formation of sulfides. Therefore, additions of organic material are expected to provide little additional benefit.

Silica encapsulation also was not retained in the final selection process. Lead is not expected to substitute for silica in the silica tetrahedron because of the large radius of the lead ion. The ionic radius of lead is considerably large than zinc and cadmium and is not geometrically compatible with this structure (i.e., will not fit). The void in the center of the tetrahedral structure is approximately 0.6 angstroms while the ionic radius of lead is approximately 1.2 angstroms. Therefore, silica encapsulation must primarily work through physical isolation and adsorption. We have already discussed adsorption. It is difficult to place a thin veneer around each particle to isolate it. Further, this thin veneer may be compromised through abrasion.

2.2 Summary of Rationale for Selecting Reagent

In general, greater bioavailability is associated with greater solubility in the acidic environment of the stomach. Compounds that form under an alkaline environment, such as carbonates and oxides, tend to be more bioavailable because they are more soluble under acidic conditions. Conversely, compounds that are stable under acidic environments, such as sulfides, tend to be less bioavailable because they are relatively insoluble in the environment of the stomach.

The bioavailability of lead phosphates is of interest because of the potential formation of lead phosphates upon application of phosphatic amendments. Lead phosphates tend to be of intermediate bioavailability (Ruby et al. 1999) except for a particular chloropyromorphite ($\text{Ca}_5(\text{PO}_4)_3\text{Cl}$) which has a low bioavailability (Ruby et al. 1999). In studies of a Missouri soil, lead bioavailability was reduced from 25-40% through application of phosphate-based soil amendments (Ruby et al. 1999). The percent reduction in bioavailability is in agreement with studies conducted in EPA Region VIII. In the studies performed by Region VIII, lead phosphates fell into the medium category with reductions in bioavailability varying from 25-75% (personal communication).

Phosphatic material also has a buffering capacity and, therefore, is effective in ameliorating acidity that may result from the oxidation of pyritic ores. Researchers at West Virginia University (West Virginia University 1988) found that apatite was effective for buffering against increases in acidity when used at 4-5 percent by weight and particle size of less than 18 mesh (1.0 mm diameter).

Others have looked at additional aspects of bioavailability and reported that apatite additions to soils have resulted in decreases in extractable lead as well as concurrent decreases in plant tissue concentrations of lead. Adding hydroxyapatite to soils decreased the lead concentrations in plant shoots by 92-98% relative to unamended soils (Traina and Laperche, 1999). Increased lead concentrations were found associated with the plant roots after adding hydroxyapatite. However, it was determined through analyses by X-ray diffraction (XRD) and scanning electron microscope (SEM) that lead phosphates, such as pyromorphite, had precipitated on the exterior root surfaces. As discussed previously, a metal may also be more or less bioavailable depending on whether it has precipitated as a solid or adsorbed onto a solid, with the adsorbed form being potentially more bioavailable (Gordon et al. 1999).

Based on literature and field studies, the general class of apatites was selected as having the most promise for success. The solubilities of various apatites were evaluated using the water composition of sample number SF271 obtained in May of 1999 (Woods 2000), along with the MINTEQA2 geochemical computer code. The water composition and code were used to

investigate the concentrations of lead maintained in solution by chloropyromorphite ($\text{Pb}_5(\text{PO}_4)_3\text{Cl}$) and hydroxypyromorphite $\text{Pb}_5(\text{PO}_4)_3\text{OH}$. Pyromorphite has been identified as forming in mine-waste contaminated soils. Further, the solubility of an hydroxyapatite ($\text{Ca}_5(\text{PO}_4)_3\text{OH}$) was investigated using the water composition of sample SF271 (Woods 2000). An hydroxyapatite was used to estimate the quantity of phosphate that would go into solution if apatite were used as a chemical reagent to immobilize metals. The results of these evaluations at varying pH values are presented in Tables 1 to 3.

Table 1
Lead Concentrations ($\mu\text{g/L}$) Maintained in Solution by $\text{Pb}_5(\text{PO}_4)_3\text{Cl}$
(Chloropyromorphite) at Varying pH Values

pH Values in units													
pH	4.0	4.5	5.0	5.5	6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	10.0
Pb ($\mu\text{g/L}$)	803	329	130	48.2	16.7	6.16	3.16	2.6	3.09	4.43	7.04	12.5	28.2

Table 2
Lead Concentrations ($\mu\text{g/L}$) Maintained in Solution by $\text{Pb}_5(\text{PO}_4)_3\text{OH}$
(Hydroxypyromorphite) at Varying pH Values

pH Values in units											
pH	5.0	5.5	6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	10.0
Pb ($\mu\text{g/L}$)	4,246	1,562	592	248	131	96.9	93.3	103.5	124.3	164.3	65.2

Table 3
Phosphate Concentrations ($\mu\text{g/L}$) Maintained in Solution by $\text{Ca}_5(\text{PO}_4)_3\text{OH}$
(Hydroxyapatite) at Varying pH Values

pH Values in units												
pH	4.0	4.5	5.0	5.5	6.0	6.5	7.0	7.5	8.0	8.5	9.0	10.0
PO_4 ($\mu\text{g/L}$)	4,864	1,159	321	98.6	31.2	9.1	2.1	0.4	0.07	0.01	0.003	0.003

These results are in general agreement with laboratory studies. As noted, the above concentrations would be expected if the particular solid being discussed were in equilibrium with a water sample (SF271) collected in May of 1999 by the U.S. Geological Survey (USGS)

(Woods 2000). Chloropyromorphite ($\text{Pb}_5(\text{PO}_4)_3\text{Cl}$) was computed to maintain lead concentrations below the primary drinking water standard of 15 $\mu\text{g/L}$ between a pH of 6.5 and 10.0. Computations assuming lead concentrations were controlled by hydroxypyromorphite ($\text{Pb}_5(\text{PO}_4)_3\text{OH}$) were also made. In these computations, the concentrations of lead were always above 15 $\mu\text{g/L}$ across the range of pH values (Table 2).

Concentrations of phosphate (PO_4) were less than 10 $\mu\text{g/L}$ at a pH value of 6.5 or higher when hydroxyapatite controlled phosphate concentrations in solution. The above tables illustrate the importance of pH when using apatite to immobilize metals such as lead. This is one of the reasons we recommend neutralizing the soil, after amendment application, to a pH value between 6.5 and 8.5. The tables also illustrate the importance of chloride (Cl^-) in the water as the lead phosphate with chloride (chloropyromorphite) achieved a lead concentration (Table 1) than the hydroxypyromorphite (Table 2).

There is a potential for use of apatite in remediation activities to mobilize arsenic. Roy et. al. (1986) observed that adsorption of arsenate was significantly reduced by the presence of phosphate. They attributed the decreased adsorption of arsenate, in the presence of phosphate, to competitive adsorption. If arsenic is a concern, other reagents, such as iron or zeolites, should be considered.

We suggest addition of chloride salts to promote chloropyromorphite formation. Candidate chloride salts include sodium chloride, and potassium chloride which is available as an industrial by-product in the study area. Divalent salts, such as CaCl_2 , also may be used. Divalent salts promote soil aggregation and improve certain soil properties (e.g., hydraulic conductivity) while the opposite can occur with application of excessive quantities of monovalent salts (sodium and potassium salts). It is doubtful that added salt concentrations will be high enough to affect soil properties. However, prior to selecting the salt to be used, a calculation will be made to assist in determining if concentrations of chloride salts are sufficiently high to merit consideration of the impact of the cationic valence state of the salt being added.

3.0 Evaluation Criteria

Reagents must be tested after treatment to determine their effectiveness. Three general categories for testing the effectiveness of a treatment option are bioavailability, leachability, and toxicity. These three categories for testing the effectiveness of various treatment applications are discussed briefly in the following paragraphs. Testing categories and procedures discussed in the following paragraphs may be used to aid in the selection of reagents or in testing the effectiveness of reagents already selected. They will be used in this study primarily to test effectiveness.

3.1 Bioavailability

Bioavailability reduction is the primary focus of these studies. Accordingly, the waterfowl studies (in vivo) will be used to test the bioavailability.

It is recommended that in vitro studies be conducted concurrently with the in vivo studies. In vitro studies use reagents that, preferably, simulate the acidity of the stomach. These reagents contact the treated material and metal concentrations are then measured to evaluate removals. The in vitro studies are less costly and time consuming than in vivo studies but only measure indirectly what in vivo studies evaluate directly. Currently, in vitro extraction tests are being validated independently at three laboratories (Ruby et al. 1999). If one can correlate the in vitro results with the in vivo results it may reduce the cost and time for additional reagent tests. It should be recognized that in vitro tests may not account for the effects of the grinding mechanisms in waterfowl digestion.

3.2 Leachability

A secondary measure of effectiveness may be leachability. Standardized batch procedures such as the Synthetic Precipitation Leaching Procedure (SPLP) and the Toxicity Characteristic Leaching Procedure (TCLP) often are used to test the effectiveness of particular reagent additions. For mining wastes not disposed in a municipal landfill, the SPLP procedure may simulate expected metal leaching more accurately than the TCLP. Therefore, the SPLP may be a more appropriate test to screen reagents or evaluate their effectiveness than the TCLP. Alternatively, basin surface or groundwater, as appropriate, may be used to measure pre- and post-treatment metal leachability. Indigenous water may provide more realistic simulations of metal leaching than standardized testing procedures (e.g., SPLP) but may not be readily comparable to results reported in the literature.

Batch experiments may be conducted as single extractions or as sequential extractions where the solution contacting the treated waste material is removed and replenished several times. Sequential batch extractions have the advantage of providing an indication of metal leachability over time.

3.3 Toxicity (Characteristic)

A tertiary measure, if needed, of effectiveness might be toxicity. As opposed to providing a representation of the leaching potential of treated waste materials, the TCLP is more suited to assist in understanding disposal issues. That is, does the treated waste exhibit the characteristic of toxicity? If so, the treated material is considered a hazardous material and must be disposed

accordingly. For example, spent apatite (from water treatment) will often exhibit the characteristic of toxicity because of zinc and cadmium concentrations (Conca 2000, personal communication).

The in vivo experiments planned as part of these studies will address toxicity via bird mortality

If plants were grown in contaminated soils and waters, phytotoxicity could be studied to determine if vegetation is improved with reagent additions.

4.0 Proposed Soil Treatability Studies

Two studies are currently proposed to assess the performance of candidate soil amendments for soils and sediments of the Coeur d'Alene River and its associated lateral lakes and wetlands. To ensure that data collected as part of these studies are of sufficient quality and quantity to support selection of effective soil amendments, the study designs should follow the EPA's Data Quality Objectives (DQO) framework (EPA 1994a). The DQO process is a systematic planning approach based on the Scientific Method for establishing criteria for data quality and for developing data collection designs.

DQOs are qualitative and quantitative statements derived from the outputs of the first six steps of the DQO Process that:

1. Clarify the Study objective
2. Define the most appropriate type of data to collect
3. Determine the most appropriate conditions from which to collect the data
4. Specify tolerable limits on decision errors which will be used as the basis for establishing the quantity and quality of data needed to support the decision

The DQOs are then used to develop a scientific and resource-effective data collection design.

The DQO Process consists of the following seven steps:

1. State the problem. Concisely describe the problem to be studied. (e.g., "Contaminated sediments have negatively impacted waterfowl in the Coeur d'Alene River. Different treatment technologies have been proposed but need to be evaluated for effectiveness.")
2. Identify the Decision. Identify what questions the study will attempt to resolve, and what actions may result. (e.g., "Are the different soil amendments being proposed effective at reducing lead (arsenic, cadmium, and zinc) concentrations in

waterfowl? Are the different soil amendments being proposed effective in reducing the leachability of arsenic, cadmium, lead, and zinc in sediments?")

3. Identify Inputs to the Decision. Identify the information that needs to be obtained and the measurements that need to be taken to resolve the decision statement. (e.g.:
 - List of proposed soil amendments (with rationale for selection)
 - Mallard duck blood lead levels
 - Pre-treatment and post-treatment sediment concentrations)
4. Define the Boundaries of the Study. Specify the time periods and spatial area to which decisions will apply.
5. Develop a Decision Rule. (e.g., "If average lead concentrations in blood samples collected from mallard ducks decrease by more than ? percent, then soil amendment A will be selected as a preferred treatment technology. If average lead concentrations in treated soil after 36 months decrease by more than ? percent, then soil amendment A will be selected as a preferred treatment technology.")
6. Specify Limits on Decision Errors. (e.g., For a 30 ft x 70 ft test plot, N number of samples should be collected using a random sample collection method to estimate average metals concentrations with a decision error rate of 1%). The DQO guidance should be consulted on procedures for determining N from a known action level and desired decision error rate.
7. Optimize the Design for Obtaining Data. Review all of the above before finalizing plans.

The two proposed studies are summarized in the following sections. Prior to the start of these studies, plans should be developed that incorporate the DQO steps and provide details on the study objectives, decision criteria, action levels, sample collection and analysis methods, reporting levels, quality assurance/quality control, data handling, and reporting.

4.1 Idaho Department of Environmental Quality Field Study

This study focuses on the arsenic, cadmium, lead, and zinc contents, and lead leachability in Coeur d'Alene River soils and sediments treated with different candidate amendments. The study is to be conducted in part with funding supplied by the Coeur d'Alene Basin Commission and is

described by Zilka (2000).

Philosophy: Remain compatible with previous study by McGeehan and Williams (1999, 2000), and with proposed new agricultural practice improvement study by F. Frutchey and M. Schlepp; show variability of field conditions.

Objectives: As stated in Zilka (2000):

Primary objective: To investigate soil amendments that reduce the bioavailability of lead in soils by the formation of low-solubility lead compounds, such as lead sulfide or lead phosphate minerals.

Secondary objective: To investigate the formation of stable heavy metal minerals in the soil so as to reduce the solubility and leachability of lead, zinc, cadmium, and arsenic into groundwater and surface water.

Summary: The field based studies will occur between late winter and early Summer 2001 at two field locations. The two amendments will be applied along the northwest side of Bull Run Lake and along the northeast side of Black Rock Slough. Access to both is available by dirt roads leading from Highway 3 at Rose Lake. The study sites are owned by the State of Idaho and managed by Idaho Department of Fish and Game personnel.

A 100 ft x 30 ft test area will be laid out at each site. Exposed vegetation (i.e., not roots) will be removed. To address spatial variability of soil metal content both vertically and horizontally, both sites will be tilled to a depth of 12 in. It is anticipated that tilling will be done, depending on soil conditions, on site using a tractor and rototiller or a track-hoe with a long arm or any other equipment that can accomplish tilling from outside the test site. Four 25 ft x 30 ft subplots (2 for amendment, 1 with lime only and 1 control) will be laid out and permanently staked at each site. IDEQ proposes to use the same sites as the FWS study (Bull Run Lake and Black Rock Slough, with Harrison Slough and Round Lake as controls).

Prior to soil amendment application, samples will be taken with a soil auger at four locations in each subplot to a depth of 12 in. These samples will not be composited, to represent the heterogeneity that remains in each plot. Each sample will be split and one set of splits will be used in the laboratory-based bioavailability (feeding) studies and one set of splits will be sent for chemical analyses. Chemical analyses will include total P, As, Cd, Pb, and Zn, TCLP extractable metals (As, Cd, Pb, and Zn), SPLP extractable metals (As, Cd, Pb, and Zn), and soil pH. Samples will be taken of water that infiltrates soil sample holes and analyzed for pH and dissolved P, As, Cd, Pb, and Zn. One of the water samples will be analyzed for major anions and cations (Ca, Mg, Na, K, Cl⁻, and SO₄²⁻). An analysis of major cations and anions can aid in an

understanding of interactions that may take place between the major cations and anions and metals of interest. For example, Ca^{2+} competes with Pb^{2+} for adsorption sites. Similarly, SO_4^{2-} can compete with PO_4^{3-} and AsO_4^{3-} for adsorption sites. Note that the set of splits sent to FWS for the feeding study will be composited at the time it is incorporated into the feed mix.

The amendments will be applied and tilled to a depth of 12 in. and allowed time to react. For each subplot, a piezometer or other equipment will be installed to allow monitoring of pore water quality. At 2 months, 12 months, 24 months, and 36 months after application, soil samples will be obtained in each plot at 8 in. depth. Chemical analyses will include the same suites as the initial samples. Water samples analyzed for major anions and cations always will be collected from the same location. The soil samples (4 per plot) will be composited by plot.

Two amendments will be mixed with Coeur d'Alene floodplain soil in the field. They are: 1) Phosphoric acid plus lime plus chloride salt, and 2) Fishbone apatite (see discussion above for the rationale). It is anticipated that the phosphoric acid will be the more effective, based on past studies. The application rate of amendments is yet to be finalized, but 1% P by weight is proposed. Only the soils amended with the first treatment will be used in the FWS feeding study, as described below (section 4.2).

In 2002:

1. Set up treatment groups to study bioavailability reductions with fishbone apatite amendment

At the end of the 3-plus year period, data from the feeding of laboratory treated soil and the field amended soil in conjunction with the chemical analysis will be compiled, analyzed, and summarized and presented in a joint report.

Measurements: The protocols for the various tests are not specified by Zilka (2000), but we have included some possible (see also section 3.0).

Soil metal contents: As, Cd, Pb, Zn, Ca, K, Mn, and Na (Method IN-CLP, IN-CLP-LOW, SW-846 6010B, AVS/SEM)

Toxicity Characteristics Leaching Procedure (TCLP) (Method SW-846 1311)

Synthetic Precipitation Leaching Procedure (SPLP) (Method SW-846 1312)

Soil pH (Method 150.1, SW-846 9045C)

Dissolved metals contents in water: As, Cd, Pb, Zn, Ca, K, Mn, and Na (Method

DIN-CLP-LOW, D200.7, SW-846 D6010B, SW-846 D7000 series for specific metals)

Water pH (Method 150.1, SW-846 9040B)

Inorganic ions in soil and water: Cl^- , SO_4^{2-} , PO_4^{3-} , HPO_4^{2-} , H_2PO_4^- (Method 300.0; chloride only: 325.3; phosphates only: 365.1, 375.1 through 375.4)

4.2 U.S. Fish and Wildlife Service (FWS) Field/Laboratory Study

This study focuses on the bioavailability of lead in Coeur d'Alene River sediments treated with phosphoric acid amendments. The study is to be conducted in collaboration with USGS, IDEQ, and EPA, and is described by Audet (2000).

Philosophy: Remain compatible with previous FWS studies by Heinz et al. (1999), and Hoffman et al. (2000); control as many variables as possible.

Objectives: To determine the bioavailability of lead to mallards in Coeur d'Alene Basin River sediments treated with phosphoric acid amendment.

Amendment: Liquid apatite (phosphoric acid) at a rate of approximately 1% phosphate, as P, by dry weight of soil. Soil pH should be raised to 7.0 or higher with lime and/or limestone if it is initially below 6.0. Chloride salt will also be added to promote the formation of chloropyromorphite. This amendment combination should be effective under all moisture regimes; lead will be permanently bound, although zinc and cadmium probably will be adsorbed and therefore may be re-released if conditions change. Note that until mineralization occurs, phosphoric acid will have a tendency to release phosphate, which may create a nutrient pollution problem. Reaction time should be at least six months, and up to one year.

Summary: One group of fully grown male mallards will serve as controls and be fed a diet containing 12% clean sediment from Round Lake. A second group will be fed a diet containing 12% lead-contaminated sediment collected from Harrison Slough in the Coeur d'Alene River. Four additional groups will be fed a diet containing 12% lead-contaminated sediment collected from Harrison Slough and treated with phosphoric acid. There will also be two additional control groups for a total of eight groups. The sediment used will be composited and homogenized, mixed with the amendments and allowed time to react; the resulting mix will then be added to commercial waterfowl diet and pelletized. The study will be conducted during cool weather, either in the fall or spring, following a 30-day period of quarantine. As an additional note, a laboratory study conducted at the Patuxent Wildlife Research Center concluded that the bioavailability and toxicity of lead-contaminated sediment is not significantly altered via the

homogenization and pelletization process employed in waterfowl feeding studies (Dan Day, 2001).

Based on the consensus of stakeholders, the following treatment groups will be included in the FWS's study.

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|----------------|---|
| Group 1 | Round Lake, controlled lab setting, no amendment |
| Group 2 | Harrison Slough, controlled lab setting, no amendment |
| Group 3 | Harrison Slough, controlled lab setting, amendment (phosphoric acid w/ lime or limestone and w/chloride salt) applied in lab and aged |
| Group 4 | Black Rock Slough, controlled lab setting, no amendment |
| Group 5 | Black Rock Slough, field test plot, collect sample after amendment (phosphoric acid w/ lime or limestone and w/ chloride salt) applied and aged in field plot |
| Group 6 | Bull Run Lake, controlled lab setting, no amendment |
| Group 7 | Bull Run Lake, controlled lab setting, amendment (phosphoric acid w/ lime or limestone and w/chloride salt) applied in lab and aged |
| Group 8 | Bull Run Lake, field test plot, collect sample after amendment (phosphoric acid w/ lime or limestone and w/ chloride salt) applied and aged in field plot |

Measurements: Protocols are those described in Heinz et al. (1999).

- Bird body weight and concentrations of lead measured in blood, liver, and kidney at 8 weeks
- Hematocrit, hemoglobin, protoporphyrin, and delta-aminolevulinic acid dehydratase (ALAD)

It is expected that the USFWS and the Patuxent Wildlife Research Center will develop and publish manuscripts presenting the study results.

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